

**Separation and Purification of Mineral Salts from  
Spacecraft Wastewater Processing via Electrostatic  
Beneficiation**

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# Separation and Purification of Mineral Salts from Spacecraft Wastewater Processing via Electrostatic Beneficiation

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Electrostatic separation is a class of material processing technologies commonly used for the sorting of coarse mixtures by means of electrical forces acting on charged or polarized particles. Most if not all of the existing tribo-electrostatic separators had been initially developed for mineral ores beneficiation. It is a well-known process that has been successfully used to separate coal from minerals. Potash (potassium) enrichment where underground salt mines containing large amounts of sodium is another use of this technology. Through modification this technology can be used for spacecraft wastewater brine beneficiation. This will add in closing the gap between traveling around Earth's Gravity well and long-term space explorations. Food has been brought on all man missions, which is why plant growth for food crops continues to be of interest to NASA. For long-term mission considerations food productions is one of the top priorities. Nutrient recovery is essential for surviving in or past low earth orbit. In our advance bio-regenerative process instead of nitrogen gas produced; soluble nitrate salts that can be recovered for plant fertilizer would be produced instead. The only part missing is the beneficiation of brine to separate the potassium from the sodium. The use of electrostatic beneficiation in this experiment utilizes the electrical charge differences between aluminum and dried brine by surface contact. The helixes within the aluminum tribocharger allows for more surface contact when being agitated. When two materials are in contact, the material with the highest affinity for electrons becomes negatively charged, while the other becomes positively charged. This contact exchange of charge may cause the particles to agglomerate depending on their residence time within the tribocharger, compromising the efficiency of separation. The aim of this experiment is to further the development in electrostatic beneficiation by optimizing the separation of ersatz and possibly real wastewater brine residues. In doing so, ideally it will yield a high potassium enrichment for use in spacecraft plant systems.

## Nomenclature

ICP-MS = Inductively coupled plasma mass spectroscopy  
*kV/in* = Kilovolts per inch

## I. Introduction

In spacecraft systems significant amounts of salts are accumulated in the wastewater brine. We believe that for dried brine residues electrostatic separation can yield concentrated potassium enrichment for use in spacecraft plant systems after further downstream processing. The goal of this study is to optimize the separation of a binary salt mixture of NaCl and KCl based on the manipulation of a verity of variables that affect the separation abilities of the electrostatic separator.

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Brine beneficiation is essential for the longevity of plant life in distant space exploration missions. Like Saline soils, brine has a high concentration of  $\text{Na}^+$ , which may result in passive accumulation of  $\text{Na}^+$  in root and shoot of plants.<sup>1</sup> High levels of  $\text{Na}^+$  can displace  $\text{Ca}^{2+}$  from root membranes, changing their integrity and thus affecting the selectivity for  $\text{K}^+$  uptake.<sup>2</sup> The possibility of high internal  $\text{K}^+$  concentrations can reduce extreme sudden environmental events like cold, frost, late season rains, high salt stresses and heat waves. Potassium act as catalysts for many of the enzymatic processes in plants that are necessary for optimum plant functions.  $\text{K}^+$  is also needed at high concentrations inside plants during early stages of its vegetative growth phase; it is a key nutrient that determines a plant's tolerance to stresses. Another major role of potassium is osmoregulation, which regulates the use of water in plants. The osmoregulation process affects water transport in the xylem, maintains high daily cell turgor pressure which affects wear tolerance, affects cell elongation for growth and most importantly it regulates the opening and closing of the stomates which affect transpirational cooling and carbon dioxide uptake for photosynthesis. Taking inconsideration the latter, it becomes essential to obtain optimum separation of  $\text{K}^+$  and  $\text{Na}^+$  in spacecraft systems.

Electrostatic separation is a dry technique in which a mixture of minerals may be differentiated according to their electrical conductivity. For potash minerals, which are not naturally conductive, the separation must be preceded by a conditioning step that induces the minerals to carry electrostatic charges of different magnitude and, if possible, different polarities. In micro electronics, theories of work functions of conducting and semi-conducting material have been well developed using Fermi level, donor and hole theories.<sup>5,10</sup> Materials with different energy levels accept electrons when in contact with a material of higher or lower work function. This transfer of electrons continues until equilibrium is reached between the Fermi energy level of the two materials. Once equilibrium is met the two materials will now have equal but opposite charges where their difference in surface work functions determine the polarity and maximum potential difference between them. Only those charges immediately surrounding the contact points are involved in the charge transfer, which is why surface structure and conditions are extremely important amongst separation. The amount of charge developed during contacts largely determines the separation efficiency.

In industry Electrostatic separation is possible because potash minerals are not naturally conductive and can only be used where gangue minerals are conductive. In Germany, researchers have developed a process for dry beneficiation of complex potash ores. Particle size, conditioning agents, and relative humidity are used to separate ore into three fractions.<sup>7</sup> The components are separated in stages, using up to four stages and the particles pass directly from one stage to another. The company that has developed this process, Kali und Salz GmbH, does not provide a detail description of the process, but do reveal the basic dimensions of a two row tube set up, 25 cm apart and 2.5 m long, at 100 kV direct current. Using this process for beneficiation, all the energy that is consumed when drying out the potash beneficiated by wet means is saved and tailings disposal problems associated with alternative wet separation processes eliminated.<sup>3</sup> Triboelectrification and electrostatic beneficiation is also a well-known technology that has been used to separator coal from minerals,<sup>4</sup> quartz from feldspar, phosphate rock from silca, and phosphorus from silica and iron ore.<sup>6</sup> In particular Electrostatic Beneficiation has been used to enrich mineral ilmenite from lunar regolith beneficiation.<sup>9</sup> This process will one day enable self-sufficient long term space explorations by providing the minerals needed to produce building material for habitats, radiation protection, landing/launch pads, as well as the oxygen and water necessary for human life.<sup>8</sup>

## II. Material/Methods

Potassium and sodium play a major role in determining the duration of plant life in long-term space explorations. The electrostatic benefactor configuration for this study was designed to achieve maximum efficiency of this separation and improve the enrichment of  $\text{K}^+$ . Initially the first 12, 10g 1:1 mole  $\text{KCl}/\text{NaCl}$  samples were grinded in a mecable for 2 minutes prior to coming in contact with the tribocharger. This was thought to be an ideal solution to its separation. Making a fine grind salt mixture increases the number of contacts between salts and lowers the mass of individual salt particles, allowing a stronger influence from electrostatic forces. The smaller particle size theoretically would also interact with the contact tube (tribocharger) more frequently resulting in a shorter residence time needed to obtain a polarity shift.

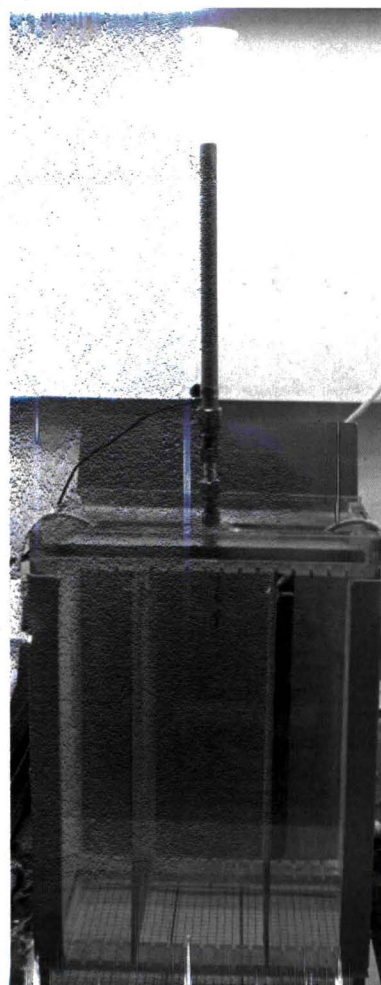
Figure 1. depicts the electrostatic benefactor configuration used for the separation. Where the tribo-charger fed into the electrostatic benefactor was 23 cm in length, with 1.27cm outer diameter. A copper wire mesh was used for

the electrode plates, which were separated 4, 6, and 7 inches apart depending on the drop. Each 10g sample of NaCl/KCl was fed at a slow rate into the tribocharger using a 10ml funnel, where Figure 2. depicts the helix aluminum structure inside the tribocharger. Aluminum was chosen as the tribocharger material because its surface work function is between that of NaCl and KCl. As the salt particles roll down the tube a charge develops on their surfaces due to the differences in the surface work functions. A series of baffles were implemented inside the tribocharger to provide the most contact with falling particles as they pass through by gravity. Prior to the particles entering the electrostatic chamber the exit point of the tribo-charger had to be tapered down in order to obtain a more uniform drop of the samples into the center-collecting bin. To obtain this the aluminum tribocharger was tapered with compression fittings ending with a 3/8 OD aluminum tube. This modification to the previous apparatus used for electrostatic beneficiation of lunar regolith allowed for separation to occur due to the charge differences between particles and the applied voltage to the electrodes vs. random disbursement.

Taping the negative and positive electrodes with a discharge rode was used prior to collecting each sample. This was done to eliminate any charge that may be held within the capacitance created by the two electrodes. Each sample tube was also weighed before and after samples where collected from the bins. This provided information needed to calculate a mass balance to determine if the mass in each sample correlated with  $K^+$  enrichment. Also it gave the basis in establishing how much of each sample was being lost after each drop and what percentage went into each sample bin.



**Figure 2.** Aluminum Tribocharger with baffles and compression fittings



**Figure 1.** Electrostatic Benification apparatus with 10ml funnel on top of tribocharger

Studies have shown that initial contact with the tribocharger will not be enough to affect the adsorbed layer associated with the contact surface of the salts particles. The greater positive charge developing on the salt particles indicates the easier escape of electron from the aluminum, which means a lower surface work function. Without a proper tribocharge, the adsorbed layer on KCl crystals will not be effected and their work function will be like that of NaCl, lower than that of aluminum: therefore, gaining a positive charge. With an efficient tribocharge, KCL will have a polarity shift, where it will then be attracted to the positive electrode. To counter this effect samples 13-20 where loaded in an aluminum tube and held on top of a mechanical agitator for 30s to super charge the particles. This allowed the salt grains to obtaining a maximum potential difference of  $7nC$ .<sup>10</sup>

### III. Results/Discussion

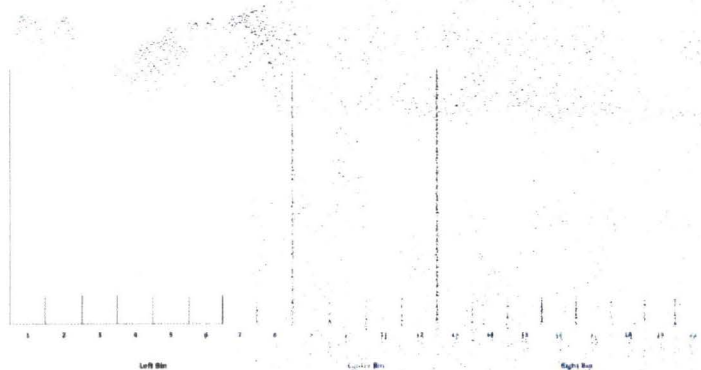
All samples where analyzed using ICP-MS, where approximately .1g from each sample was diluted to 20ppm. Preliminary trial observations suggested that the binary salt mixture would give a fan like spread once a high enough voltage was applied to the system. This then lead to our experimental factorial where the physical

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**Table 1.** Factorial sheet used to determine parameters for optimum  $K^+$  enrichment. Samples 17,18, and 20 showed the most promising results for  $K^+$  enrichment.

Run	Plate distance(in)	Total Voltage(kV)	kV/in	%humidity	brine	Bins	Grind	Notes
1	7	26000	3714	0	KCL/NaCl	9;12	fine	dried/ fine grounded
2	7	26000	3714	room	KCL/NaCl	9;12	coarse	Control/fine grounded
3	7	13000	1857	0	KCL/NaCl	9;12	fine	low Voltage/fine grounded
4	7	6500	929	0	KCL/NaCl	9;12	fine	low Voltage/fine grounded
5	7	26000	3714	0	KCL/NaCl	9;12	fine	fine grounded
6	4	14860	3715	0	KCL/NaCl	9;12	fine	medium distance/fine grounded
7	7	19500	2786	0	KCL/NaCl	9;12	fine	fine grounded
8	7	26000	3714	0	KCL/NaCl	9;12	fine	fine grounde/no tribocharger
9	7	26000	3714	0	NaCl	9;12	fine	NaCl only/fine grounded
10	6	22300	3717	0	KCL	9;12	fine	KCl only/fine grounded
11	6	22500	3750	0	KCL/NaCl	9;12	fine	medium Distance/fine grounded
12	6	26000	4333	0	KCL/NaCl	9;12	fine	medium Distance/fine grounded
13	6	5570	928	0	KCL/NaCl	9;12	fine	supper chargged
14	4	14860	3715	0	KCL/NaCl	9;12	fine	supper chargged
15	4	7430	1858	0	KCL/NaCl	9;12	fine	supper chargged
16	4	3710	928	0	KCL/NaCl	9;12	coarse	supper chargged
17	6	26000	4333	0	KCL/NaCl	9;12	coarse	supper chargged
18	4	26000	6500	0	KCL/NaCl	9;12	coarse	supper chargged
19	4	13000	3250	0	KCL/NaCl	10;11	coarse	supper chargged
20	4	26000	6500	0	KCL/NaCl	8;13	coarse	supper chargged



**Figure 4.** Schematic of bottom try collecting bins. The negative and positive electrodes were placed near the left and right collecting bins respectively.

#### A. Enrichment and Yield Calculations

Equations 1 and 2 were used to determine the yield and enrichment factors of analyzed data from the ICP-MS. The enrichment and yield calculations gave the basis in defining what changed parameters had the most influence on separation. If there were an increase in enrichment then there would be a decrease in yield or vice versa depending on the on the parameter that's being altered.

$$\text{Enrichment} = \frac{\frac{\text{KCl-effluent}}{\text{NaCl-effluent}}}{\frac{\text{KCl-influent}}{\text{NaCl-influent}}}$$

$$\text{Yield} = \frac{\text{KCl-effluent}}{\text{KCl-influent}}$$

(1) (2)

#### B. Normal Charge vs. Super Charge

The initial results from the first 12 drops in Fig 5. implied that we were not allowing enough contact time between the tribocharger and the falling particles. The adsorbed layer associated with the contact surface on KCl was not being affected resulting in minimum separation. As seen in Table 2, addressing this phenomenon in samples 13-20 improved  $K^+$  enrichment by 67%. Super charging proved to be an effective means of optimization in the separation process.

separation process.

### C. Electrode Plate and Sample Bin Placement

The electrode plate distance and placement of the dividers in the sample bins played a role in the  $K^+$  yield and enrichment factors as well. To minimize the variables in our factorial we chose to leave the sample dividers in the same positions of slots 9 and 12 for majority of the drops. Doing this allowed us to focus on more dominant variables in the intent that we could optimize separation without adding unwanted variables to the factorial. Adjusting the distance between the electrodes to four inches was

found to be the most consistent in separation at high voltages. From Figs 6 and 7 it is evident that changing the position of the sample bin dividers affected potassium's yield and enrichment in the left collecting bin. The smaller the area of the left sample bin the higher it's yield do to its ability to capture more particles being directly attracted towards the positive electrode.

Table 2.  $K^+$  enrichment in normal and super charged drops.

Sample Bin	Drop (1-12)	Drop (13-20)
L	1.046	2.211
C	0.875	1.699
R	0.934	0.39
	Normal Charge	Super Charge

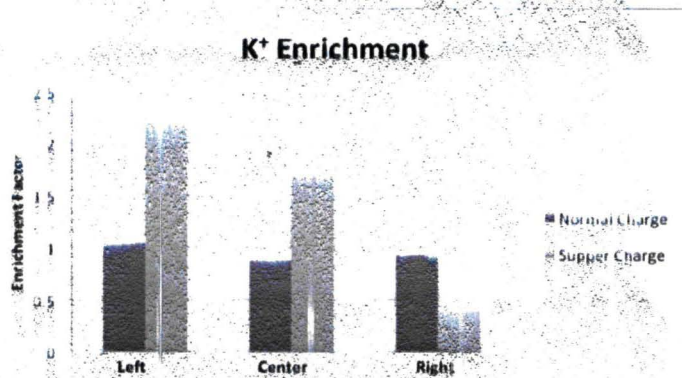


Figure 5. Super charged particles in the left collecting bin showed the most improvement in  $K^+$  enrichment.

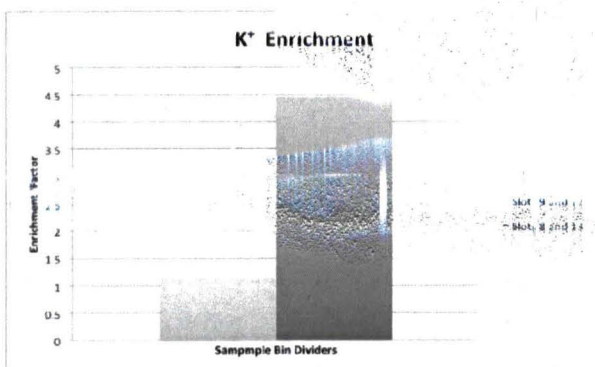


Figure 6. Electrode plate distance of four inches was used when changing sample bin dividers. Dividers spread furthest apart gave the largest  $K^+$  enrichment.

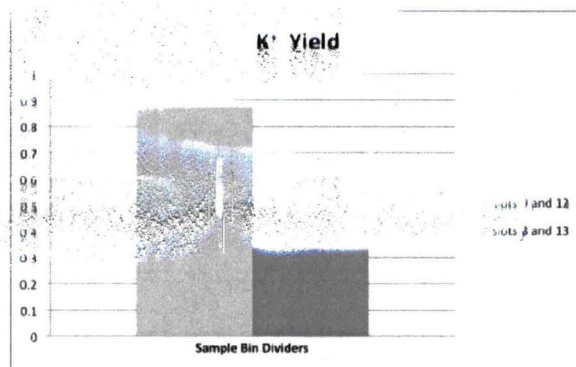


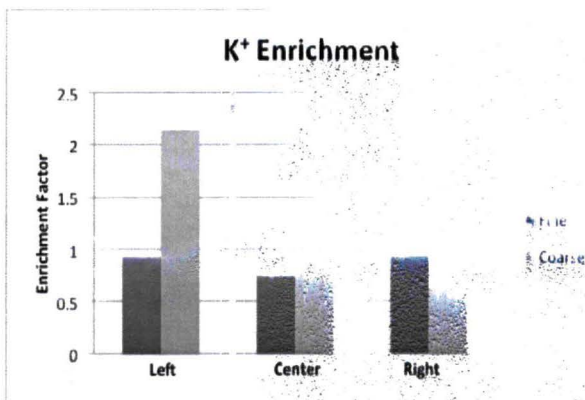
Figure 7. Implies that potassium's yield is much higher when the sample bin dividers are closer together.

### D. Coarse Grind vs. Fine Grind

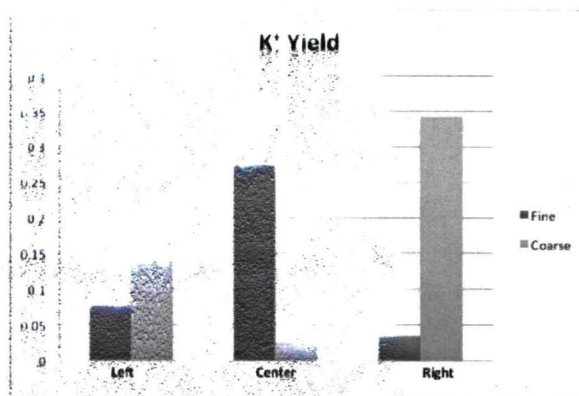
Surface structure and particle size both play a role in the separation abilities of the salt crystals. Although results indicate that moving the electrodes closer together may increase the possibility to improve KCl enrichment, salts that are fine or coarse grind had an influence on separation as well. There is a possibility that some of the salt



plate distance of 7 inches at a high voltage (26000kV) shows that the coarse grind separation was more efficient than that of the fine grind.



**Figure 8.** Particle size proved to have an effect on  $K^+$  enrichment in the left collecting bins.



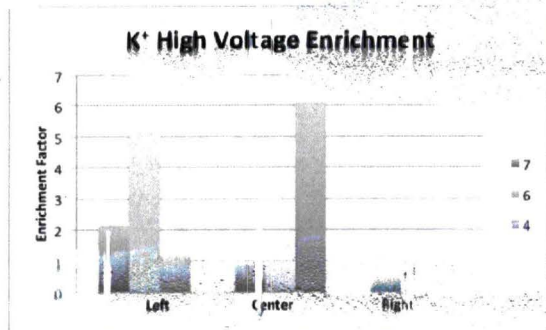
**Figure 9.** Shows a high yield of  $K^+$  in the center bin for fine grind samples, which implies that majority of the particles may have agglomerated before entering the electrical field.

#### E. High Voltage vs. Low Voltage

Samples were dropped between 3710kV and 26000kV. In theory the maximum voltage that can be held below the electrical arc voltage should have the best separation do to the attracting forces from the opposing charge differences in the salt crystals. This gave a minimum and maximum volts per inch of 3250kV/in and 3715kV/in respectively. Figures 10 and 11 indicate that at maximum and minimum voltage, electrode plate distances of 7 and 6 inches will have the most enrichment of  $K^+$  in the left bins. This improvement in potassium implies that there is an influence from electrostatic forces based on voltage input.

**Table 3.** The sample bin placement for all drops where in the 9 and 12 slots.

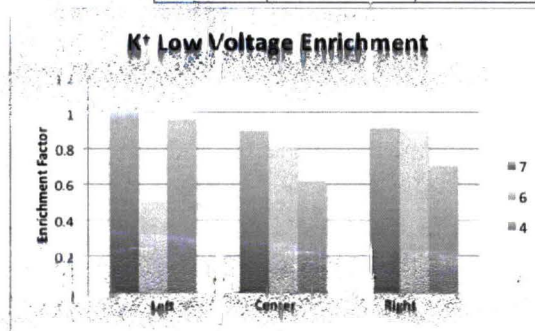
Electrode Distance	K <sup>+</sup> Enrichment factor at High Voltage		
7	2.142	0.771	0.625
6	5.358	1.55	0.001
4	1.146	6.1	0.001
	Left	Center	Right



**Figure 10.** Drops with an electrode plate distance of 4 inches showed a high enrichment in the center do to the negative electrode being closer to the center bin.

**Table 4.** At low voltage there was very minimum influence from electrostatic forces.

Electrode Distance	K <sup>+</sup> Enrichment factor at Low Voltage		
7	1.062	0.898	0.915
6	0.504	0.808	0.897
4	0.963	0.618	0.703
	Left	Center	Right



**Figure 11.** At a low voltage the enrichment of  $K^+$  in the center bin was decreased in drops with plate distances of four inches because the electrical forces were not strong enough to deflect the particles back towards the center bin.

## F. Mass Balance

Data from ICP-MS was used to conduct a mass balance of each sample, where on average, each sample had a 19% lost in influent. Data in Table 5. shows the total mass from each sample drop along with the amount being lost. Majority of the lost was caused by the entrapment of salt particles within the tribocharger and sample bins. Tables 6. shows the yield lost after each drop, where the contact exchange of charges in some cases caused the particles to agglomerate forcing them to accumulate within the tribocharger and in the cracks of the collecting bins.

**Table 5.** Drop 6 showed an increase in mass do to the accumulation of particles in the tribocharger from previous drops.

Drop	Total Mass	Mass Lost
1	7.867	2.133
2	9.019	0.981
3	9.321	0.679
4	9.39	0.61
5	2.903	7.097
6	13.672	-3.672
7	8.365	1.635
8	5.071	4.929
9	6.129	3.871
10	7.735	2.265
11	8.873	1.127
12	7.911	2.089
13	8.994	1.006
14	8.207	1.793
15	9.127	0.873
16	9.382	0.618
17	9.001	0.999
18	8.869	1.131
19	8.835	1.165
20	8.786	1.214

**Table 6.** Yield lost after each drop

Drop	Na <sup>+</sup>	K <sup>+</sup>
1	0.256	0.163
2	0.355	0.493
3	0.476	0.080
4	0.873	0.878
5	0.510	0.612
6	0.319	0.288
7	0.633	0.771
8	0.383	1.000
9	0.973	0.091
10	0.607	0.584
11	0.520	0.403
12	0.650	0.579
13	0.325	0.475
14	0.311	0.353
15	0.302	0.427
16	0.335	0.410
17	0.269	0.429
18	0.312	0.479
19	0.214	0.378
20	0.282	0.324

## IV. Conclusion and Recommendations

In a tribo-electrostatic separation process, the greater the charge potential the better a separation is obtained. Therefore, the separation in this experiment was improved by controlling the amount of contact time between the salt particles and the tribocharger. The samples super charged in aluminum tube prior to entering into the electrostatic separator showed a significant improvement in the enrichment of K<sup>+</sup> in the left bins. While fine grind samples ran at a high voltage where forced to the electrode with too much force resulting in an accumulation of salt on the electrode, in the wrong sample bin, and on the wall behind the electrodes.

To further the development in the separation on KCl and NaCl through Electrostatic beneficiation samples should be super charged prior to being dropped in the electrostatic benefactor, this would allow for the particles to reach a maximum charge difference, achieve a polarity shift, and optimize enrichment. Another factor to consider is gravitational forces. Running experiments in low gravity will mimic gravitational separation conditions in spacecraft systems. This will increase the residence time of each particle being dropped within the electrostatic benefactor. Increasing residence time enables more influence from electrostatic forces, hence improving K<sup>+</sup> enrichment. Broadening the scope in electrostatic beneficiation will close the loop in using recovered soluble nitrate salts from wastewater brine residues for plant fertilizer. This novel approach will aid in the successful advancement in outer orbit long-term space explorations.



orbit long-term space explorations.

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